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FUNDAMENTALS OF LIQUID PROPELLANT SENSITIVITY

AF 04(611)-11207

Allen J. Tulis and Ted A. Eriksen
IIT Research Institute

TECHNICAL PROGRESS REPORT IITRI-C6085-8

Air Force Rocket Propulsion Laboratory
Research and Technology Division
Air Force Systems Command
Edwards, California

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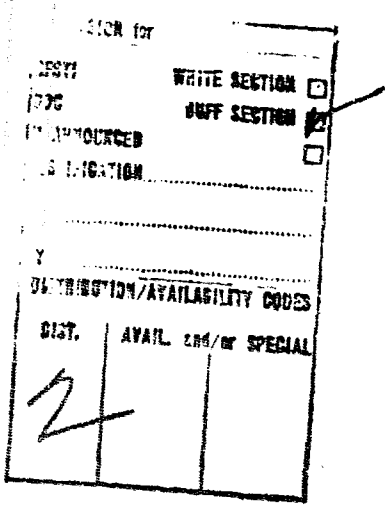

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FOREWORD

This is Report No. IITRI-C6085-8, the second Technical Progress Report on IIT RI Project C6085, Contract No. AF-04-(611)-11207, entitled "Fundamentals of Liquid Propellant Sensitivity." This report covers the period from 1 March through 31 May 1966 and is submitted in July 1966.

This program is being conducted by IIT Research Institute, 10 West 35th Street, Chicago, Illinois 60616, for the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Edwards Air Force Base, California, under Air Force Program Structure No. 750G, AFSC Project No. 3148, and AFSC Task No. 3148-0-1. The program monitor for this project is Ralph Fargnoli, 2nd Lt., USAF/RPCL, of Edwards Air Force Base, California.

The program is under the direction of Mr. Ted A. Erikson, Senior Chemist. The experimental work is being performed by Dr. James Keith , Research Chemist; Mr. Theodore Burgwald, Research Chemist; Mr. Allen J. Tulis, Associate Engineer; and Mr. Odis Flynn, Technician. Other IITRI personnel who have contributed to the program include Dr. Morton J. Klein, Director of Applied Chemistry Research; Dr. Irvine J. Solomon, Senior Chemist; and Mr. Robert I. Brabets, Research Engineer.

Data included in this report are recorded in Logbooks C16606, C16616, C16617, and C16996.

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

This technical report has been reviewed and is approved:

George F. Babits, Lt, Colonel USAF
Chief, Propellant Division

ABSTRACT

In tests with Compound T, using nitrogen driven gas shocked to reflected temperatures about 1150°K, the corresponding changes in time for induction delay and sample consumption for increases in reflected-shock pressure from 500 to 1500 psia were 400 to 50 and near zero to 500 microseconds, respectively. Although the induction time decreases at higher pressure the decreased consumption rate indicates a relatively slow-burning that is an improvement in explosion sensitivity. A vaporization mechanism which appears to be inhibited by high external pressure was proposed to be a rate-controlling step in the explosion transition of most CNF type compounds that were studied to date. In additional experiments, explosions were simulated by spark discharges so as to verify certain characteristics of the light and pressure sensors as used in shock-tube sensitivity testing of cryogenic compounds. Efforts to characterize the vaporization of inert freons were only partly successful.

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SECTION I

INTRODUCTION

In this program, the approach to propellant densensitization is based on a fundamental evaluation of the various parameters that affect initiation, growth, and propagation of explosive CNF compounds.

Cryogenic shock-tube testing procedures[1] were developed during a previous program (under Contract No. AF 04(611)-9566). In this program, during 1965 extensive testing was conducted on Compounds R, T, H, PFG, and PFF. Specific parameters used were reflected-shock temperature, pressure, and composition of the driven gas in the shock tube. Time-delay measurements of light output and work output thus identified sensitization aspects of these compounds. Sample characteristics, such as size, area, surface geometry, and temperature, influenced the results and were evaluated, and reported in the Annual Summary Report.[2]

Three potential stages that contribute to the explosive process are of concern in this program. They are (1) fast vaporization, (2) homogeneous vapor reaction, and (3) heterogeneous surface reaction. The shock-tube technique induces combinations of these stages to occur at and near the surface of the condensed phase by energy interactions.

Time-delay measurements themselves cannot determine the sensitivity of explosives. They merely identify a starting point and an end point without regard to events in the interim, during which both physical and chemical processes occur. Either process determines not only rate, but perhaps events; that is, they determine whether the explosive will explode, burn, decompose, or merely vaporize.

Interpretation of the light-output and work-output responses of CNF and other compounds tested by the shock-tube technique is progressing, and many valuable interpretations have been

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[1] Erikson, T. A., "Fundamentals of Liquid Propellant Sensitivity," Annual Summary Report, Contract No. AF 04(611)-9566 conducted by IIT Research Institute, Chicago, Illinois, Dec. 15, 1964.

[2] Erikson, T. A. and Tulis, A. J., "Fundamentals of Liquid Propellant Sensitivity," Annual Summary Report, Contract No. AF 04(611)-9566 conducted by IIT Research Institute, Chicago, Illinois April 1965.

achieved to date. Preliminary results, particularly those for Compounds R, T, and H, are reported.

Interpretation necessitated calibration of instrumental response characteristics. The major portion of the experimental program has involved verification, calibration, and interpretation of response from the light and the pressure sensors. To accomplish this (1) spark-discharge simulated explosions, (2) inert-compound fast (flash) vaporizations, and (3) lead azide detonations were tested in the shock tube. Major results of these studies also are reported.

SECTION II

STATUS OF RESEARCH

1. EXPERIMENTAL FACILITY AND PROCEDURE

a. Shock Tube

Shock-tube testing procedures for CNF compounds are discussed elsewhere.[2] Several freons were shock-tube tested during this report period. These were selected because of their physical properties of boiling point, critical temperature and pressure, and heat of vaporization, which are similar to those of the CNF compounds under consideration. The freons were tested as liquids, ice, and snow depositions, with nitrogen, argon, and helium as driven gases.

Nitromethane and lead azide also were tested, nitromethane for its vaporization and explosion characteristics and lead azide for its detonation characteristic.

Spark-discharge simulated explosions were conducted under static and dynamic conditions in the shock tube. For static tests, the discharge electrodes were placed at the sample site, and instrumentation was set up as for CNF shock-tube testing.

For most purposes, spark-discharge energy was controlled by pressure in the shock tube although the spark gap also was varied to optimize desirable discharge characteristics. Discharge occurred when the voltage built up sufficiently to jump the spark gap. Therefore no "switching" losses occurred. Triggering of the oscilloscope was tested for variation between different modes, i.e., voltage discharge, light initiation, pressure output, and transient voltage induced upon the instrumentation from the oscillatory discharge of the capacitor. Except for the variations noted later, variation was negligible.

The dynamic tests were unsuccessful to date because of the difficulty in properly sequencing the spark discharge with the shock condition.

b. Instrumentation

Pressure-output responses from the Kistler gauge were monitored with and without a two-stage LC filter and the charge amplifier. Pertinent aspects of these hook-ups are reported.

Light-output responses from the light-sensor apparatus were monitored directly to the scope. Different ranges of sensitivity and response of the light sensor were evaluated. In spark tests, the voltage transient induced in the instrumentation from the

capacitor discharge required heavy grounding of the scope and related components, shielding of the components, short leads, floating discharge, and other means to calibrate successfully.

2. LIGHT-SENSOR RESPONSE

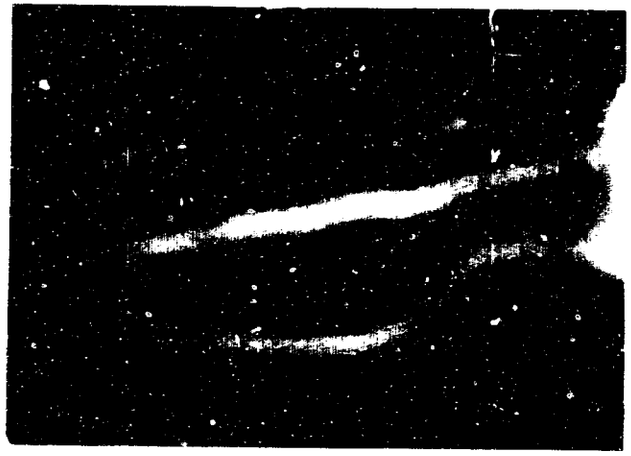
a. Calibration

The light sensor being used is a Texas Instruments Type LS-400 N-P-N planar silicon phototransistor. It was selected because of its high sensitivity, fast response, and suitability for simple and rugged application to the shock-tube study. In our circuit design, five selectable series (load) resistors of 1K, 3K, 10K, 30K, and 100K ohms were incorporated, with the highest resistance maximizing sensitivity and the lowest resistance maximizing response characteristics. Since rise time in all cases was <1 μsec and our primary concern in the shock-tube study of CNF compounds to date has been the time to the initial generation of light, the highest sensitivity mode was utilized in all CNF tests. However, recent calibration established that for further analysis, including response to cessation of light (fall time), the fast response mode (with associated decreased sensitivity) is more informative.

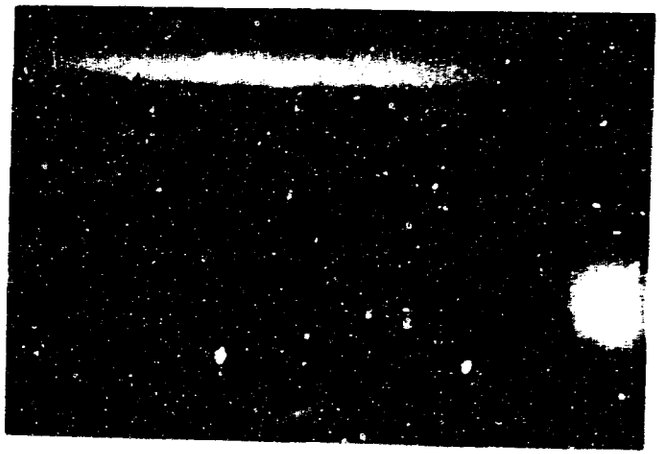
Oscillograph L-1 (Figure 1) illustrates the sensitivity of the light sensor to a spark-simulated explosion, triggered by the electrical discharge of the capacitor. The sweep speed was 0.2 μsec/cm. The time to full saturation was 0.75 μsec, with a delay of 0.4 μsec to initial light output.

Oscillograph L-2 (Figure 1) illustrates the rise times to the light outputs from the spark-discharge simulated explosions for both the highest sensitivity mode and the highest response mode. Twenty-five tests for each mode or a total of fifty tests, are superimposed on this oscillograph. The sweep was 0.1 μsec/cm, and the traces were triggered by the light output. Approximately 0.25 μsec were required for rise time to full saturation.

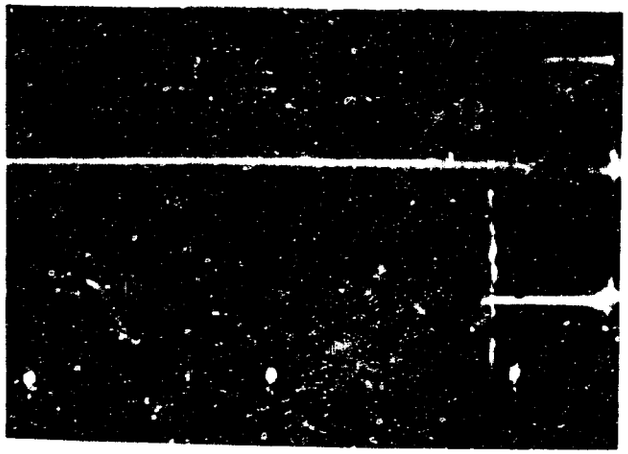
Oscillograph L-3 (Figure 1) illustrates the fall time of the five sensitivity modes selectable. Five tests for each mode, or a total of twenty-five tests, are superimposed on this oscillograph. The sweep speed was 10 μsec/cm, and the transient signal caused by the oscillatory discharge of the capacitor triggered the oscilloscope. The fast fall time (about 2 μsec) was in the least sensitive mode and progressed successively to the slowest fall time (about 100 μsec) in the most sensitive mode. The high sensitivity mode follows declining light or infrared radiation to a greater degree than the less sensitive mode. The bottom trace on this oscillograph is the work-output signal from the pressure transducer, which registered the 25 corresponding "rings" to the spark-discharge simulated explosions.



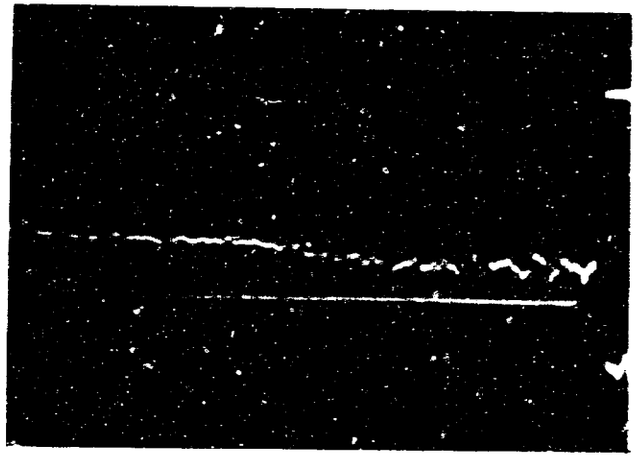
Test L-1: static test.
Sweep speed: 0.2 μsec/cm.
Top trace: discharge voltage.
Bottom trace: light response.



Test L-2: static test.
Sweep speed: 5 μsec/cm.
Light response at five superimposed mode settings.

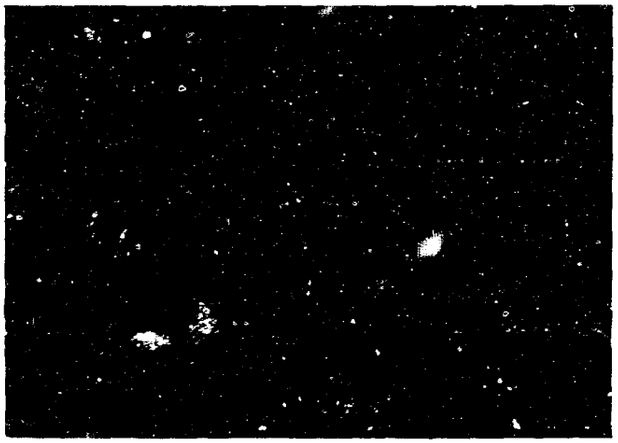


Test L-3: static test.
Sweep speed: 10 μsec/cm.
Top traces: light response.
Bottom trace: pressure response.

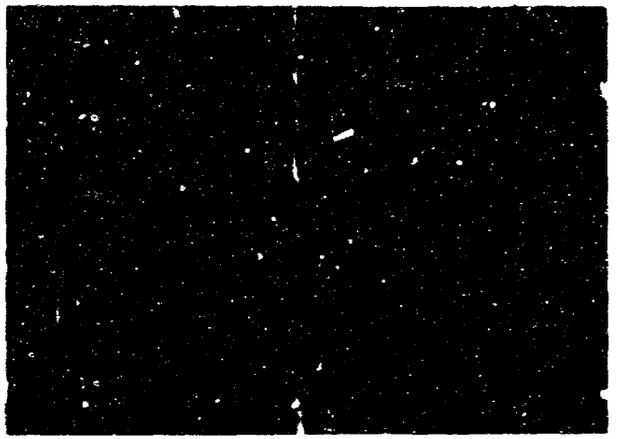


Test L-4: blank test with argon.
Sweep speed: 50 μsec/cm.
Top trace: light response.
Bottom trace: pressure response.

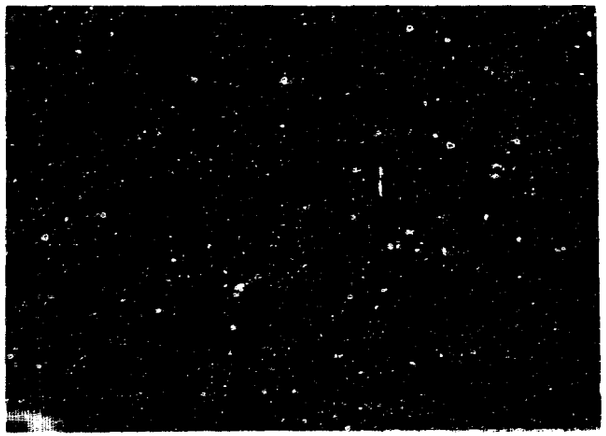
Figure 1. LIGHT-SENSOR RESPONSE CHARACTERISTICS



Test L-5: shock-tube test with Freon 11.
Sweep speed: 50 μsec/cm.
Top trace: light response.
Bottom trace: pressure response.



Test L-6: static test.
Sweep speed: 2 μsec/cm.
Top trace: discharge voltage.
Bottom trace: light response.



Test L-7: shock-tube test with lead azide.
Sweep speed: 50 μsec/cm.
Top trace: light response.
Bottom trace: pressure response.

Test L-8: shock-tube test with wetted lead azide.
Sweep speed: 50 μsec/cm.
Top trace: light response.
Bottom trace: pressure response.

Figure 1---Concluded

b. Interpretation

The light sensor has served excellently for the qualitative sensing of light output. No attempt has been made at quantitative analysis, and none is feasible with regard to intensity since it depends on a multitude of variables, both experimental (size of sample, location of light source, etc.) and instrumental (variable response to the frequency of radiation, etc.). However, several general characteristics of the light-output signal are pertinent.

Time to initial light output, time to full saturated light output, duration of light output, inconsistencies in saturated output, and time to complete declination of light output have been of recent interest. Cursory calibration and interpretation of these were conducted and are complete. Simply, a fast pulse-type light output with negligible duration is associated with an explosive reaction, while a gradual (or fast) light output of extended duration can be attributed to either (1) a nonexplosive fast-to-moderate chemical reaction, such as burning, or (2) highly heated products of reactions that radiate infrared long after the light output of the chemical reaction ceases. The subsequent oscillographs of figure 1 indicate the proper interpretation of the light-output signal.

Oscillograph L-4 (Figure 1) illustrates the light-sensor response to infrared radiation from a highly heated gas, i.e., a blank test with argon as the driven gas. The sweep speed was 50 μsec/cm, the reflected-shock temperature was about 2200°K, and the reflected-shock pressure was about 700 psia.

Oscillograph L-5 (Figure 1) is identical to L-4, except that an inert compound, Freon 11, was deposited at the sample site as snow at about -160°C. The endothermic effect of fast vaporization of Freon 11 is identified by the comparison to light-sensor response in L-4.

Oscillograph L-6 (Figure 1) illustrates the light-sensor response to a spark-discharge simulated explosion. The bottom trace is the light-output signal in the fast-response mode and had a duration of about 10 μsec; the sweep speed was 2 μsec/cm. The upper trace is the discharge-voltage monitor, attenuated 1000:1 and indicating that discharge was completed in 5 μsec. The light output was reduced to zero at 12 μsec, indicating a fall time of 7 μsec.

Oscillograph L-7 (Figure 1) illustrates the light-sensor response to the detonation of lead azide in the shock tube. Light output from a detonation of this size sample of lead azide (about 0.1 mm thick) is certainly <1 μsec (detonation velocity:

5.1 mm/μsec). It is evident from the light-sensor response in the fast response mode that light output was indeed of short duration. The sweep speed was 50 μsec/cm. The pressure-sensor calibration indicated simultaneous light and work outputs.

Oscillograph L-8 (Figure 1) illustrates the light-sensor response to fast-reacting (wetted) lead azide in the shock tube.

3. PRESSURE-SENSOR RESPONSE

a. Calibration

The pressure sensor being used is a Kistler PZ-6 miniature pressure transducer. This gauge has a resonant frequency of about 100 kHz, a characteristic termed a "ring" when brought on suddenly by the passage of sharp pressure transients. This characteristic detects the moment of explosion in shock-tube testing of explosive compounds. Calibration and interpretation of this signal have been conducted to ascertain the application of this response to the initiation scheme of explosive processes, especially in conjunction with the light output.

Spark-discharge simulated explosions at the sample site within the shock tube were utilized (1) to characterize the origin of pressure-sensor response and (2) to calibrate the intensity of the response with energy. Three capacity levels, 0.1, 1.0, and 52 mfd, covering an energy range from 0.01 to >500 joules, were used.

Figure 2 illustrates the ring-response amplitude relationship to energy at a spark-discharge capacitance of 1.0 mfd and an oscillatory capacitor discharge of 200 kHz. The relationship is linear if the discharge frequency remains constant. Results from tests at the other two capacity levels were similar, except as shown in Table 1.

Table 1

PRESSURE-SENSOR RING RESPONSE
TO SPARK-DISCHARGE SIMULATED-EXPLOSION ENERGY
AT 0.1, 1.0, and 52 mfd

Capacity, mfd	Discharge Frequency, kHz	Discharge Duration, μsec	Average Discharge Energy/Sensor Output, joules/mv
0.1	1000	7	0.12
1.0	200	16	0.42
52.0	20	90	1.00

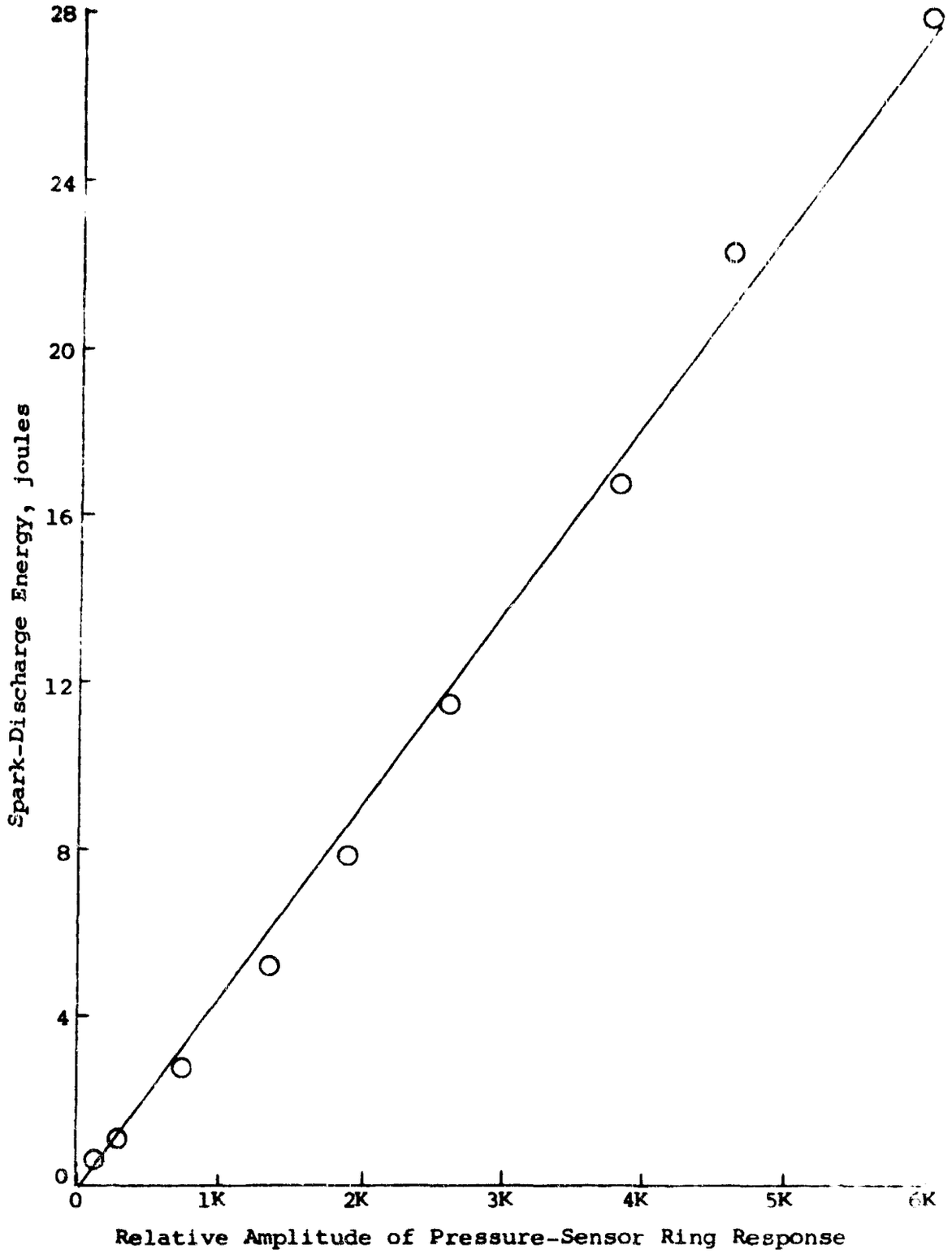


Figure 2. PRESSURE-SENSOR RING RESPONSE TO SPARK-DISCHARGE SIMULATED-EXPLOSION ENERGY AT 1.0 mfd

Figure 3 illustrates the rate-of-energy variation with the ring response amplitude. As the rate of energy release in the spark discharge increases, less energy is required for the same level of response on the pressure sensor. In Figure 3, the duration of discharge is inversely proportional to the rate of energy applied to the spark discharge when response per unit energy is the inverse of the ordinate, joules/mv.

Oscillograph P-1 (Figure 4) illustrates the light and the pressure sensors' responses to a spark-discharge simulated explosion. The top trace is the light-sensor response in the high-sensitivity mode and the bottom trace is the pressure-sensor response. The sweep speed was 50 μsec/cm. and shows the ring response delayed at about 25 μsec.

Oscillograph P-2 (Figure 4) illustrates the effect of incorporating an LC filter into the pressure-sensor circuit. The top trace is the light-sensor signal in the fast-response mode. The two-stage LC filter was designed for cutoff above 60 kHz with an attenuation of 100 at 130 kHz, the ring frequency. Therefore the actual pressure wave form of the shock wave upon the pressure sensor was evident. It was the result of continuous wall reflections of the strong sonic wave (resulting from the initial shock wave) and had a frequency of about 20 kHz. This secondary "resonant" frequency has been observed in many shock-tube tests of explosive compounds in the frequency range of about 5 to 30 kHz depending upon shock-tube parameters.

Oscillograph P-3 (Figure 4) illustrates (1) light-sensor response (top trace), (2) voltage monitor of capacitor discharge (middle trace), and (3) pressure-sensor response (bottom trace). The sweep rate was 5 μsec/cm. Discharge was complete after 5 μsec, the light output was instantaneous, and the pressure output lagged by about 20 μsec. This was the transit time for the sonic wave traveling from the sample site to the gauge. In oscillograph L-7 (Figure 1) the light output is at 120 μsec, while the work output starts at 140 μsec, indicating a 20-μsec pressure-output lag for the probably simultaneous events.

b. Interpretation

The ring response is caused by the passage of a sharp pressure transient past the pressure sensor. The shock tube-created shock wave travels down the tube and causes the pressure sensor to ring, as evidenced in all shock-tube pressure-sensor traces. Upon explosion of the compound, the shock wave causes the gauge to ring much more violently. Because of the proximity to the sensor and the near point-source of this explosion, the secondary resonant frequency is much more evident. However, the degenerated-to-

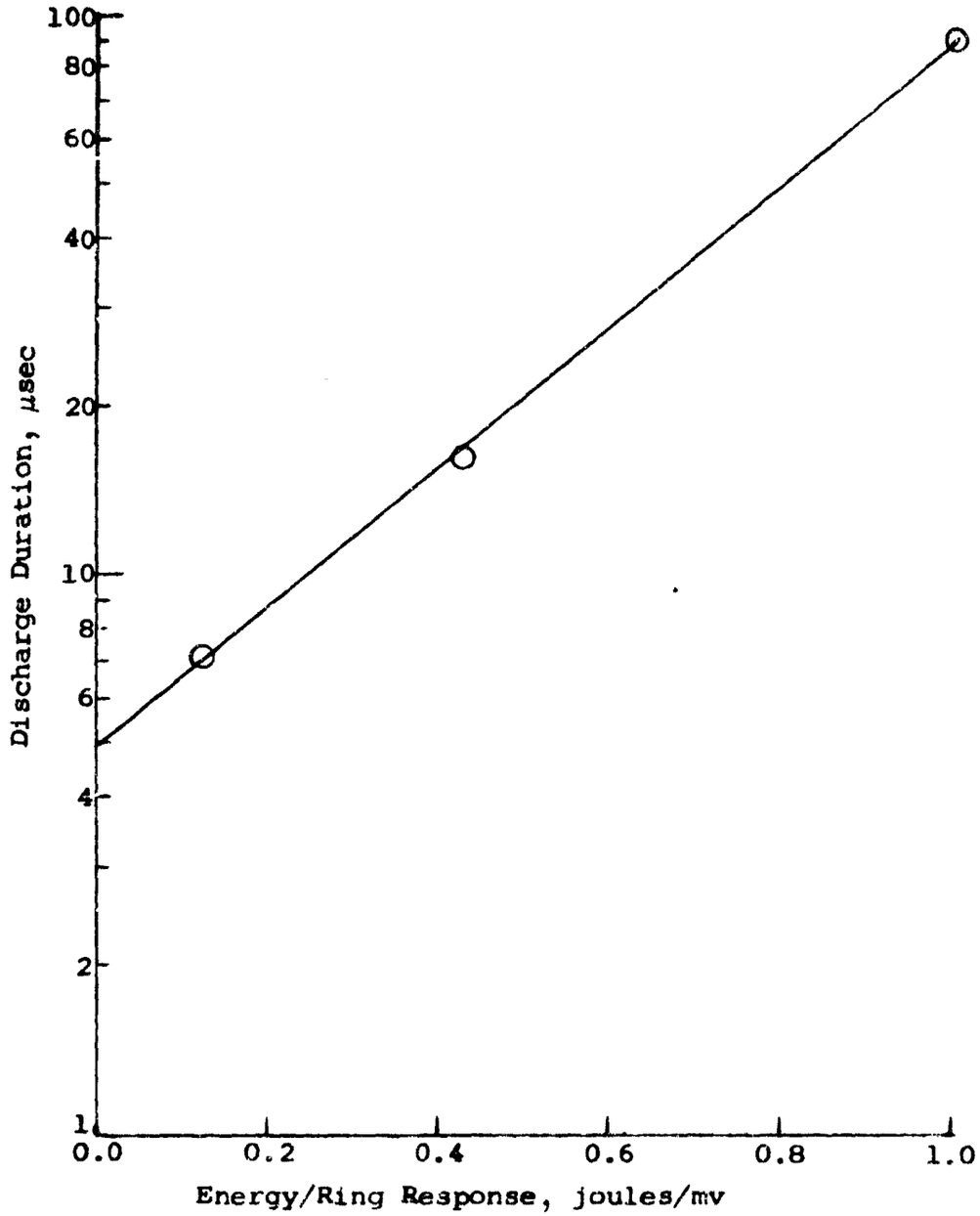
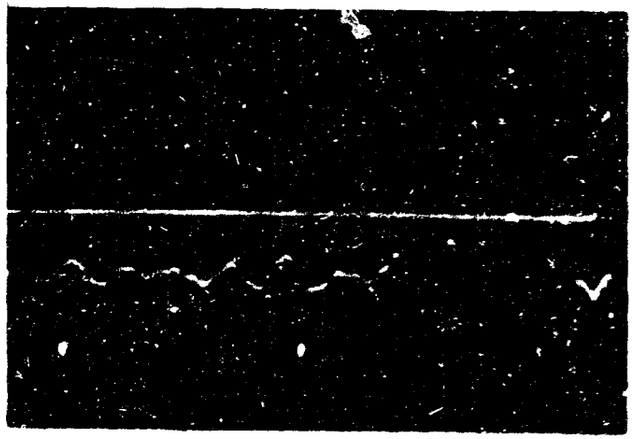
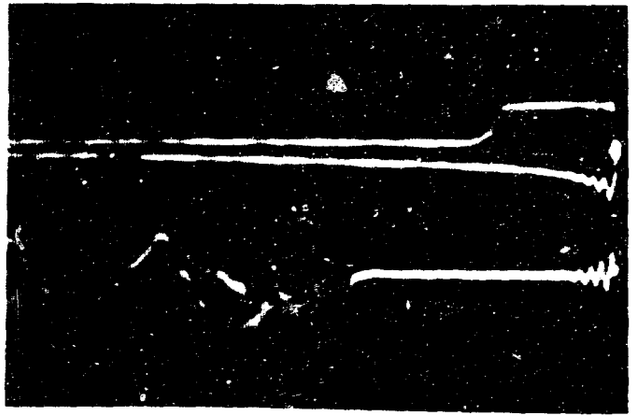


Figure 3. RATE-OF-ENERGY VARIATION WITH RING-RESPONSE AMPLITUDE

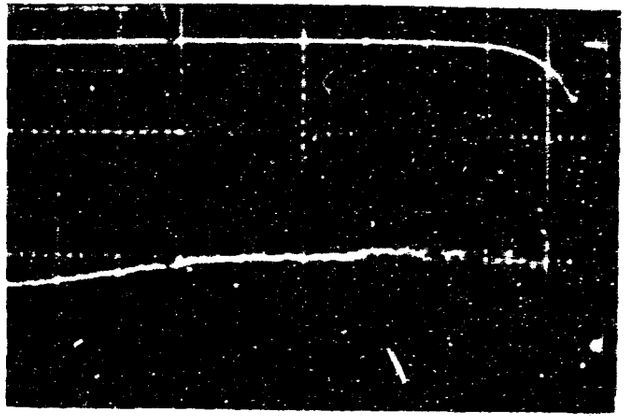
Test P-1: static test.
Sweep speed: 50 μsec/cm.
Top trace: light response.
Bottom trace: pressure response.



Test P-2: static test.
Sweep speed: 50 μsec/cm.
Top trace: light response.
Bottom trace: pressure response with LC filter.



Test P-3: static test.
Sweep speed: 5 μsec/cm.
Top trace: light response.
Middle trace: voltage discharge.
Bottom trace: pressure response.



Test P-4: shock-tube test
with gas-phase Compound PFG.
Sweep speed: 100 μsec/cm.
Top trace: light response.
Bottom trace: pressure response.

Figure 4. PRESSURE-SENSOR RESPONSE CHARACTERISTICS

sonic shock wave does travel back up the tube to the top and down again and can be picked up second, third, or more times, traveling at sonic velocity within the shock tube.

Flash vaporization preceding explosions of the CNF compounds has been investigated. A pressure-sensor response to flash vaporization was investigated. Several Freons and other inert materials were tested in the shock tube as liquids and as solid snow depositions. Confirmative evidence of work-output response to flash vaporization was not observed.

Oscillograph P-4 (Figure 4) illustrates a gas-phase explosion of Compound PFG in the shock tube. The sample was deposited at -175°C, allowed to warm to 0°C (as a gas), and tested. The work output and a sharp light output are evident. The sweep speed was 100 μsec/cm, the reflected-shock temperature was 1050°K, and the reflected-shock pressure was about 515 psia.

4. DATA INTERPRETATION AND RESULTS

The calibration test procedures establish that an explosive process at the sample site generates a ring response on the Kistler gauge through the gas phase and is directly related to energy release at the sample site. Absence of ring response denotes absence of an explosive process. Light-out duration is directly related to chemical reaction duration; i.e., pulse-type light output is associated with an explosive process whereas long-duration light output is associated with burning (slow) reaction. Although long-duration light output indicates a burning reaction, an associated ring response suggests additional phenomena such as flash vaporization, partial-sample explosion, gas-phase chemical reactions, etc.

Most CNF data collected to date have been evaluated and general interpretations and results are reported below.

Table II is a tabulation of all experimentally adequate tests conducted on Compound H. These are grouped into four general reflected-shock pressures--about 500, 700, 1100, and 1300 psia. Reflected-shock temperatures and time-delay measurements have been correlated[2] and conform to adiabatic treatment. Of greatest significance is the fact that as initiation conditions become more severe, the results show a form of desensitization; i.e., instead of exploding, a compound simply burns.

Figure 5 contains oscillographs representative of each group in Table II. For tests in which the reflected-shock pressure was increased the explosiveness was diminished. This suggests that a flash vaporization occurred and was responsible for explosion. Flash vaporization is achieved when the sample

Table II. SHOCK-TUBE TEST RESULTS FOR COMPOUND H

Test No.	T_{DT}, [a] °K	T_{RS}, °K	P_{rs}, psia	Sample Type	Total Number of Tests	Number of Tests				Time Delay (t_t), μsec	
						E[b]	EB[c]	B[d]	NG[e]	Light	Work
447	100	1100	500	Liquid	4	1	0	0	3	850	860
409	100	1200	700	Liquid	22	14	0	0	8	515	535
456	100	1300	1100	Liquid	2	0	0	2	0	120	---
457	100	1400	1300	Liquid	2	0	0	2	0	75	---

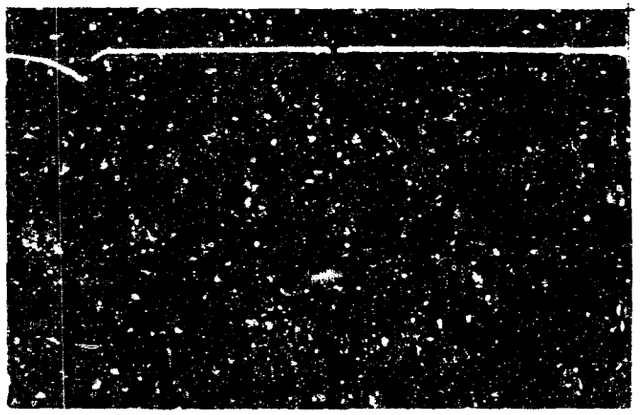
[a] Temperature of deposition and testing.

[b] Exploded.

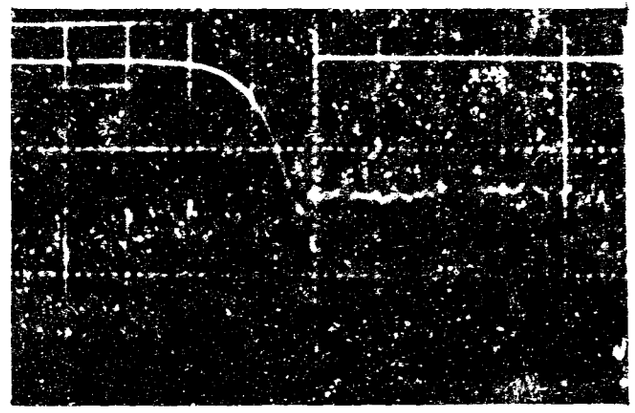
[c] Exploded and burned.

[d] Burned.

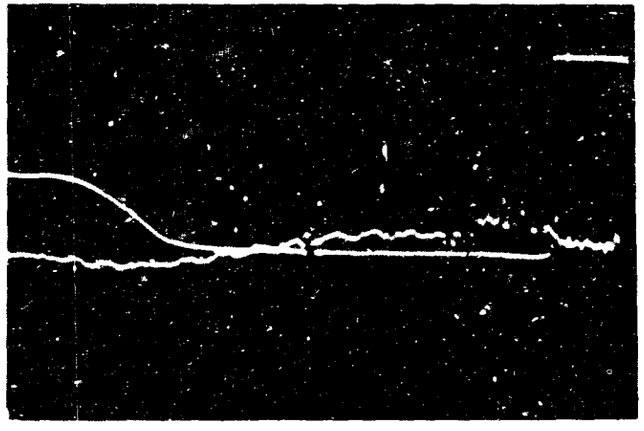
[e] Nc response for both light and work.



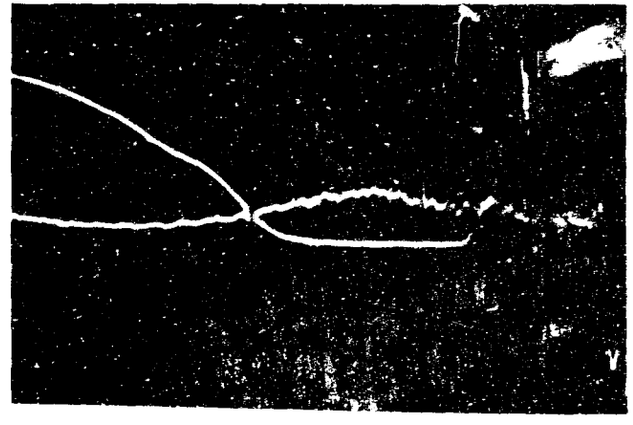
Test 447: explosion.
P_{RS} = 475 psia.
T_{RS} = 1220°K.
t_+ = 850 μsec.
Pressure trace off scale except for ring (severe) at far left.



Test 409: explosion.
P_{RS} = 710 psia.
T_{RS} = 1240 °K.
t_+ = 500 μsec.



Test 456: burning.
P_{RS} = 1115 psia.
T_{RS} = 1270 °K
t_+ = 130 μsec.
Slight pressure response.



Test 457: burning.
P_{RS} = 1305 psia.
T_{RS} = 1390 °K.
t_+ = 110 μsec.

Figure 5. COMPOUND H EXPLOSION-TO-BURNING TRANSITIONS WITH PRESSURE

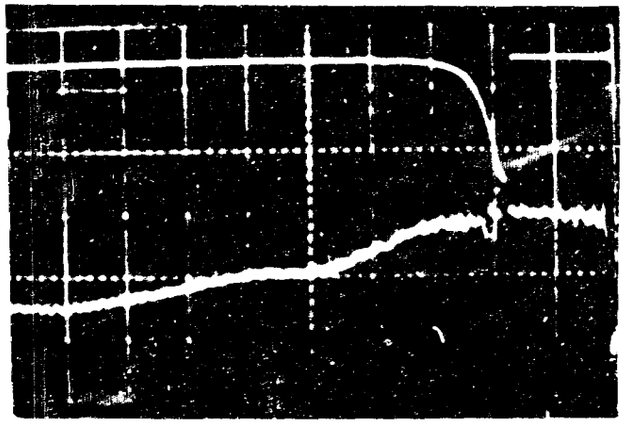
surface attains a temperature at which the associated vapor pressure is greater than the reflected-shock pressure. Conversely, if the ambient pressure is greater than the vapor pressure flash vaporization cannot take place and this type of explosion response is not possible.

The combination of large mass transfer (flash vaporization) into the hot reflected-shock gas and the instantaneous (near) gaseous chemical reaction results in growth and propagation to explosion of the condensed-phase compound. If mass transfer occurs but the subsequent chemical reaction is slow and/or not highly exothermic, explosion does not occur. Since the CNF compounds have high vapor pressures and fast chemical reactivities at low temperatures, conditions are optimized for such high-sensitivity characteristics. Nitromethane and nitroglycerin, on the other hand, require much higher temperatures before they attain similar rates of vaporization and chemical reaction. Furthermore, the low heat of vaporization of the CNF compounds also is favorable for high sensitivity with this interpretation.

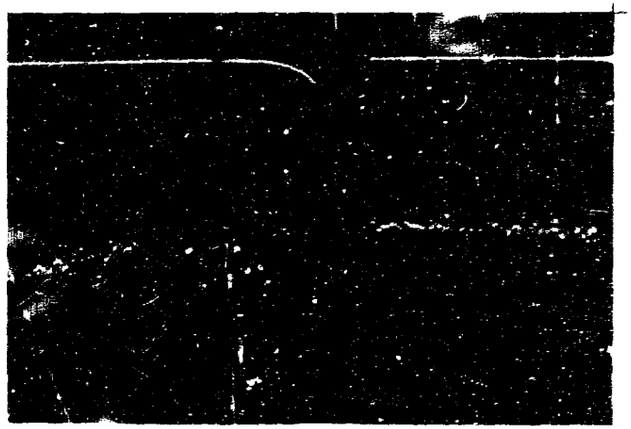
Table III is a tabulation of all experimentally adequate tests conducted on Compound T. Figure 6 contains oscillographs representative of four of the groups in Table III. These results conform to those for Compound H, except Compound T tests involved additional parameters. Compound T was tested as a solid condensed-phase sample and deposited as a snow, an ice, and degrees of both.

Examination of Table III shows that as shock conditions of reflected-shock temperature and pressure increased, the general results were in the sequence: (1) no-go, (2) explosion, and (3) burning. The shift for the snow deposition tests was slower than for the ice tests; i.e., at reflected-shock pressures of about 700 psia, the ice (cycled and noncycled) tests shifted into burning reactions, whereas the snow tests were still exploding. Because of surface geometry, the snow tests expose more surface and thus attained higher vaporization rates. Since Compound T has a relatively low critical pressure (estimated at 300 psia), the interpretation based on flash vaporization involves other considerations. The fact does remain, however, that in both ice and snow tests at reflected-shock pressures over 1000 psia, explosion was nearly precluded. In addition the exceptions, those that explode in Table III at pressures over 1000 psia can be explained (though not verified) by the probability of the samples being wet.

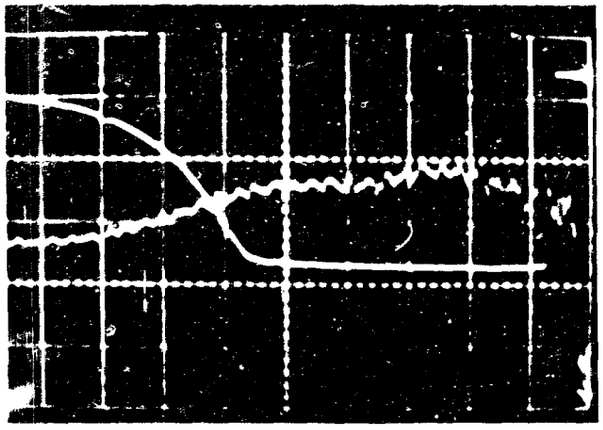
Of all the CNF compounds tested, Compounds PFF and R were most readily desensitized by increasing pressure, so that explosion-to-burning transition occurred at reflected-shock pressures under 500 psia. In the case of compound PFF, all



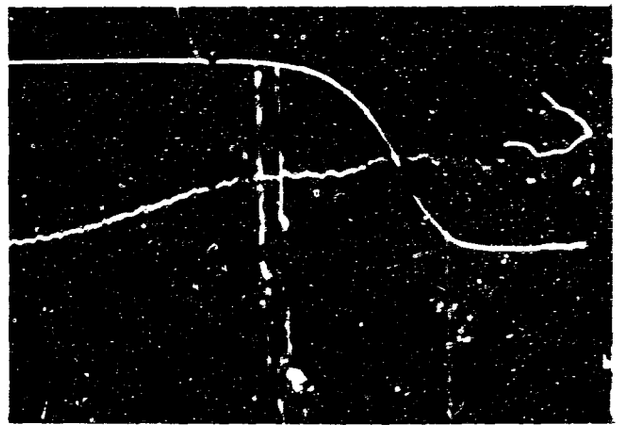
Test 329: explosion.
P_{RS} = 505 psia.
T_{RS} = 1100°K.
t_+ = 360 μsec.
Deposition as ice.



Test 308: explosion.
P_{RS} = 515 psia.
T_{RS} = 980°K.
t_+ = 415 μsec.
Deposition as snow.



Test 322: burning.
P_{RS} = 1530 psia.
T_{RS} = 1150°K.
t_+ = 65 μsec.
Deposition as ice.



Test 355: burning.
P_{RS} = 1620 psia.
T_{RS} = 1000°K.
t_+ = μsec.
Deposition as snow.

Figure 6. COMPOUND T EXPLOSION-TO-BURNING TRANSITIONS WITH PRESSURE

Table III. SHOCK-TUBE TEST RESULTS FOR COMPOUND T

Test No.	T_{LT}, °K	T_{RS}, °K	P_{RS}, psia	Sample Type	Total Number of Tests	Number of Tests				t_+, μsec	
						E	EB	B	NG	Light	Work
307	100	850	400	Snow	1	0	0	0	1	---	---
329	200	1000	500	Ice	3	1	0	0	2	360	365
308	100	1000	500	Snow	4	3	0	0	1	335	345
313	200	1200	700	Ice	8	0	6	2	0	165	200
342	100[a]	1150	750	Ice	13	0	0	11	2	145	---
303	100	1200	700	Snow	6	6	0	0	0	45	55
326	200	1150	1200	Ice	1	0	1	0	0	60	80
322	200	1100	1550	Ice	5	1	0	4	0	80	80
355	100	1100	1550	Snow	11	4	0	7	0	20	30

[a]Cycled (warmed) to 200°K after deposition and prior to testing at 100 °C.

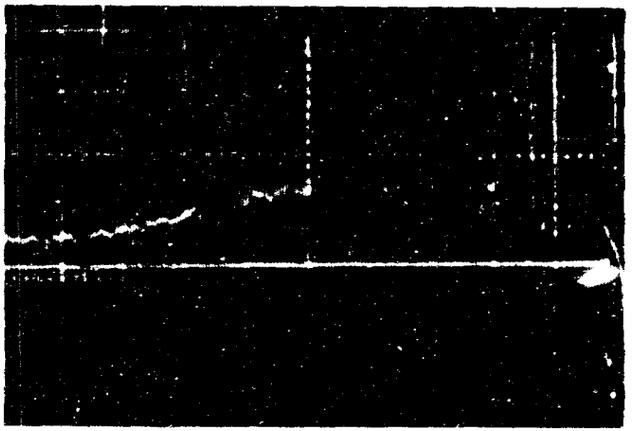
tests were conducted with a reflected-shock pressure of about 700 psia. Light outputs were of long duration, but (1) liquid PFF resulted in explosion pressure-sensor outputs, whereas (2) solid PFF resulted in questionable or very mild work outputs. In the case of Compound PFG, also tested at 700 psia, (1) it always exploded as a solid and (2) it burned with explosive work outputs as a liquid. Figure 7 illustrates these effects.

Although testing of Compound R has been most extensive, the majority of earlier tests were not experimentally acceptable because the major portion of our experimental trouble-shooting and sophistication has concerned this compound. Consequently, only the mose recent tests on Compound R are considered in this evaluation even though the earliest tests generally conformed to the interpretation.

Interpretation of Compound R results is more complex because additional parameters were evaluated. Table IV and Figure 8 illustrate the results with helium as the driven gas. Table V and Figure 9 illustrate the results with argon as the driven gas. Although these data correlate well to high pressure desensitization of explosive response independently, they do not correlate when combined; i.e., other factors, such as collision frequency and thermal capacity, which depend on gas composition, are pertinent.

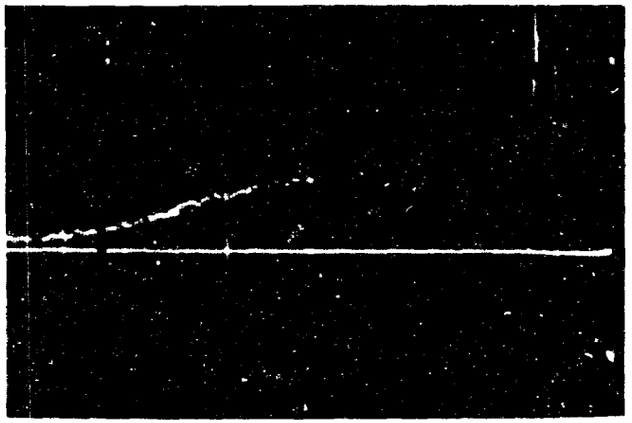
When evaluating Compound R on a qualitative basis during last year's program, it was discovered that water sensitizes this compound. Table VI and Figure 10 illustrate this sensitization, which occurred both in (1) decreased time delay to explosion and/or burning and (2) transition from burning (dry Compound R) to explosion (wet Compound R). The results are quite conclusive even though the tests were not quantitative The wet tests generally consisted of placing a hot wetted paper towel inside the closed environment of the depositing sample. The normal tests consisted of taking normal precaution to avoid moisture by using a plastic bag to maintain a positive pressure of dry nitrogen throughout sample deposition, although generally some "frost" still formed before deposition commenced; the frost was wiped away. The dry tests consisted of using phosphorus pentoxide to dry the shock tube prior to testing; the helium carrier gas, supposedly dry originally, was passed through molecular sieves.

It was evident in most cases of suspected moisture sensitization and in nearly all cases of definite moisture contamination (see Figure 10) that both light- and pressure-output signals contained an oscillation at about 30 kHz. The combination of endothermic water vaporization and exothermic Compound R reaction could result in the "sputtering" response obtained.

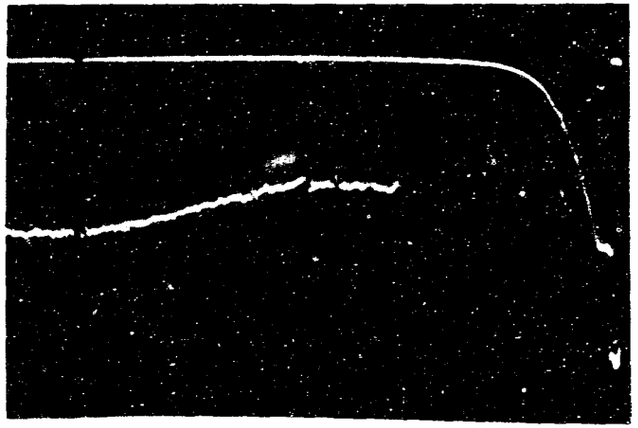


Test 407 with liquid Compound PFF: explosion and burning.
Sweep speed: 100 μsec/cm.
P_{RS} = 720 psia.
T_{RS} = 1210°K.
t_+ = 10 μsec.

Test 444 with solid Compound PFF: burning.
Sweep speed: 100 μsec/cm.
P_{RS} = ~690 psia.
T_{RS} = ~1260°K.
t_+ = 250 μsec.



Test 476 with liquid Compound PFG: explosion and burning.
Sweep speed: 100 μsec/cm.
P_{RS} = 735 psia.
T_{RS} = 1180°K.
t_+ = 10 μsec.

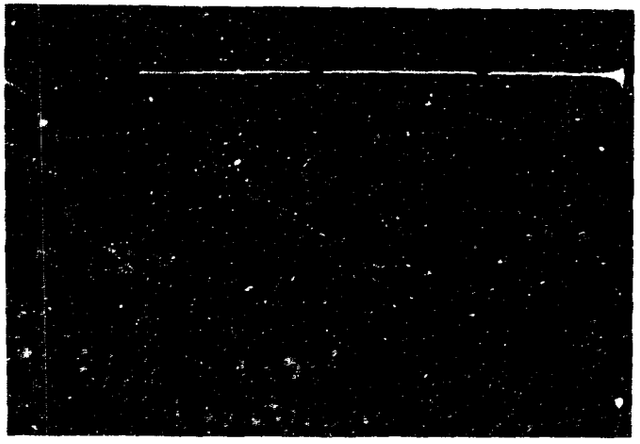


Test 461 with solid Compound PFG: explosion.
Sweep speed: 100 μsec/cm.
P_{RS} = 715 psia.
T_{RS} = 1220°K.
t_+ = 20 μsec.

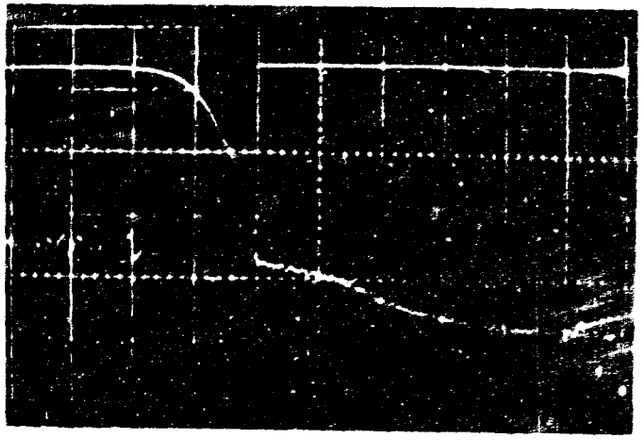
Figure 7. COMPOUNDS PFF AND PFG EXPLOSION-TO-BURNING TRANSITIONS WITH PHASE

Table IV. SHOCK-TUBE TEST RESULTS FOR COMPOUND R WITH HELIUM AS THEIR DRIVEN GAS

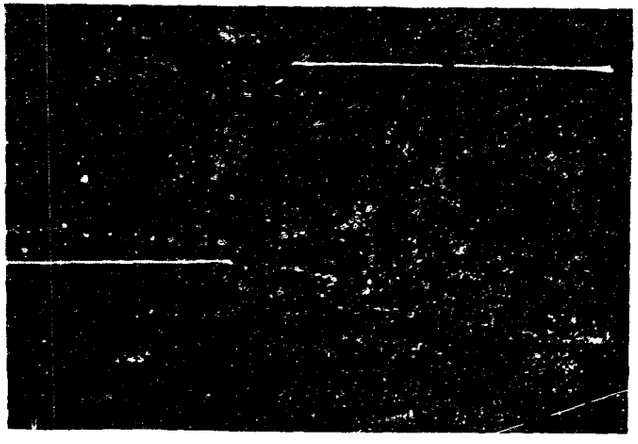
Test No.	T_{DT}, °K	T_{RS}, °K	P_{RS}, psia	Sample Type	Total Number of Tests	Number of Tests				t_+, μsec	
						E	EB	B	NG	Light	Work
267	100	900	170	Solid	1	1	0	0	0	800	805
259	100	950	190	Solid	5	4	1	0	0	615	620
268	100	1000	205	Solid	2	0	2	0	0	695	745
266	100	1100	255	Solid	2	0	2	0	0	280	285



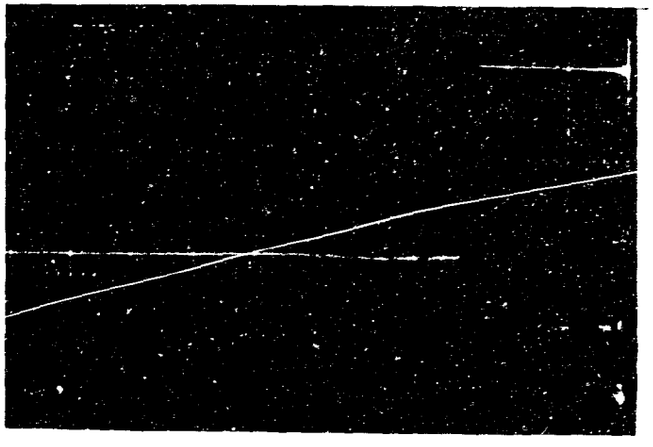
Test 267: explosion.
P_{RS} = 170 psia.
T_{RS} = 890°K.
t_+ = 800 μsec.



Test 259: explosion
P_{RS} = 190 psia.
T_{RS} = ~950°K.
t_+ = 615 μsec.



Test 268: explosion and burning.
P_{RS} = 205 psia.
T_{RS} = 980°K.
t_+ = 540 μsec.

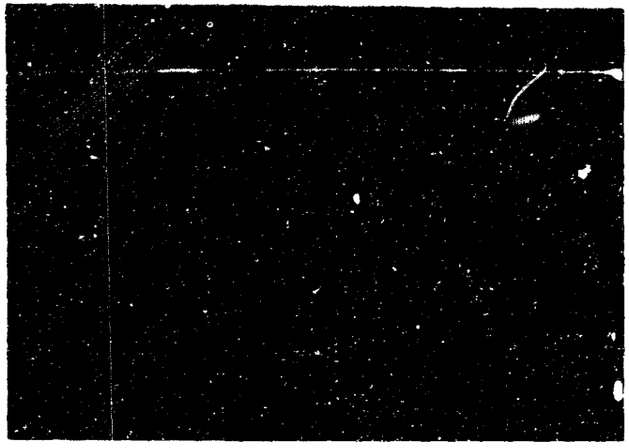


Test 266: explosion and burning.
P_{RS} = 255 psia.
T_{RS} = 1120°K.
t_+ = 260 μsec.

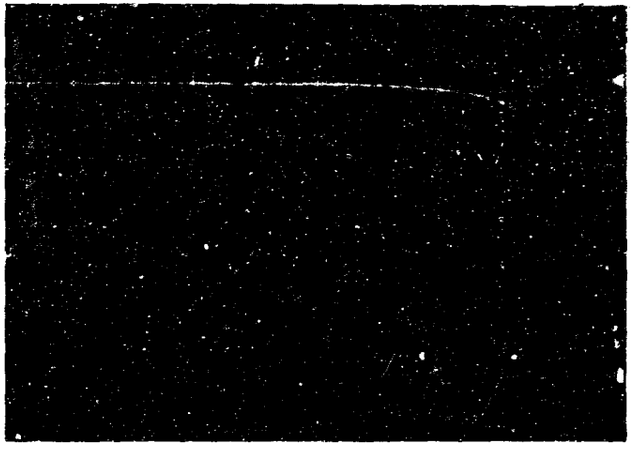
Figure 8. COMPOUND R EXPLOSION-TO-BURNING TRANSITIONS WITH PRESSURE AND HELIUM AS THE DRIVEN GAS

Table V. SHOCK-TUBE TEST RESULTS FOR COMPOUND R WITH ARGON AS THE DRIVEN GAS

Test No.	T_{DT}, °K	T_{RS}, °K	P_{RS}, psia	Sample Type	Total Number of Tests	Number of Tests				t_i, μsec	
						E	EB	B	NG	Light	Work
257	115	1500	400	Solid	2	1	0	0	1	650	660
254	115	1750	500	Solid	4	3	1	0	0	25	30
249	100	2050	650	Solid	10	0	3	7	0	80	30



Test 257: no go.
P_{RS} = 415 psia.
T_{RS} = 1490°K.



Test 254: explosion.
P_{RS} = 510 psia.
T_{RS} = 1730°K.
t_+ = 20 μsec.

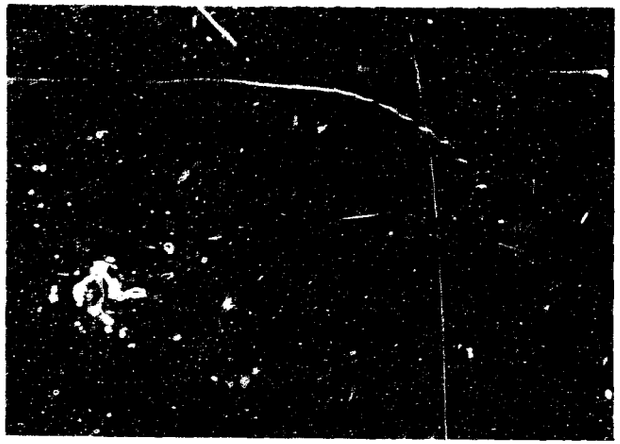
Test 249: burning.
P_{RS} = 650 psia.
T_{RS} = 2010°K.
t_+ = 130 μsec.

Figure 9. COMPOUND R EXPLOSION-TO-BURNING TANSITIONS WITH PRESSURE AND ARGON AS THE DRIVEN GAS

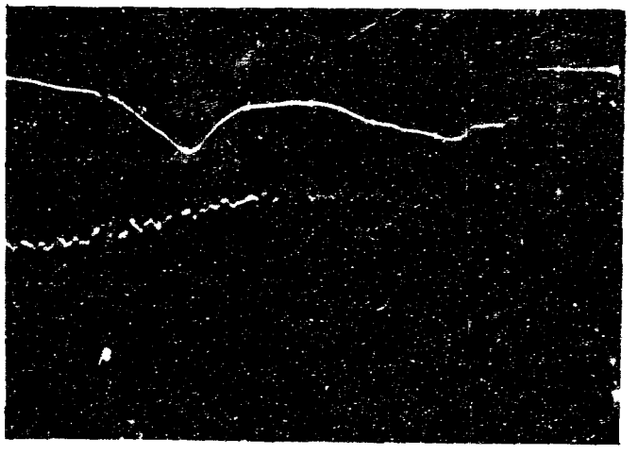
Table VI. SHOCK-TUBE TEST RESULTS FOR MOISTURE-SENSITIZED COMPOUND R

Test No.	T_{DT}, °K	T_{RS}, °K	P_{RS}, psia	Sample Type[a]	Total Number of Tests	Number of Tests				t_+, μsec	
						E	EB	B	NG	Light	Work
278	100	1200	730	Wet solid	8	4	4	0	0	90	100
239	100	1170	735	Normal solid	9	2	7	0	0	155	165
296	100	1185	730	Dry solid	13	0	7	6	0	175	165

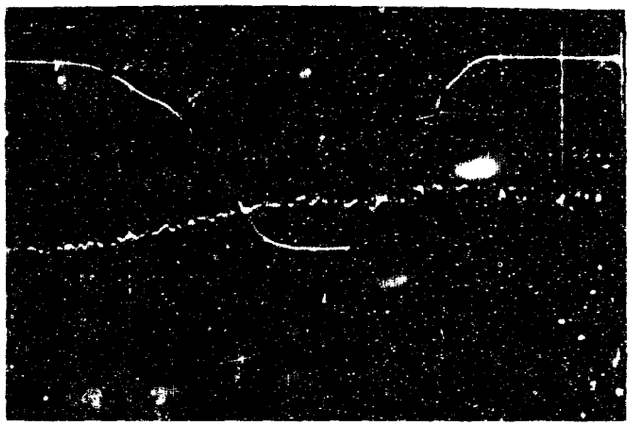
[a]See text for description of wet, normal, and dry sample-types.



Test 278 with wet Comppudd R: explosion.
P_{RS} = 735 psia.
T_{RS} = 1170°K.
t_+ = 100 μsec.
Oscillation on both wave forms.



Test 239 with normal Compound R: explosion and burning.
P_{RS} = 735 psia.
T_{RS} = 1180°K.
t_+ = 145 μsec.



Test 296 with dry Compound R: burning.
P_{RS} = 735 psia.
T_{RS} = 1170°K.
t_+ = 230 μsec.

Figure 10. MOISTURE SENSITIZATION OF COMPOUND R

SECTION III

SUMMARY AND CONCLUSIONS

Most calibrations and interpretations of both light and pressure sensors have been completed. The pertinent aspects of the instrumental-response characteristics have been presented. By its duration, the light-output sensor differentiates between a fast explosive reaction and a slow burning reaction. The pressure-output sensor identifies explosive processes by the sonic transients that are characteristic of these processes. Flash vaporization was not yet experimentally identified, but its effects are indicated in tests with inert compounds.

Of great significance is the examination of combined light and pressure responses to shock-tube initiated samples. At high reflected-shock pressures, CNF compounds gave long-duration light outputs and mild or nonexistent pressure outputs, contrary to the short-duration light and severe pressure outputs obtained at low reflected-shock pressures. Spark-discharge simulated explosions and lead azide detonations within the shock tube substantiated the interpretation that light output and ring amplitude are directly related to the energy release. Hence, slow (burning) reactions are identified by long light durations and mild ring amplitudes of pressure. The converse responses identity fast (explosive) reactions.

It is a major result of this report period that although more severe shock-tube conditions of reflected-shock pressure (at constant reflected-shock temperature) reduce the time delay to sensor response; this event is usually a burning, or low energy release as indicated by long duration light output. Conversely, the longer time delays obtained at low reflected-shock pressures generally lead to explosions. The import is that there is evidence that both physical and chemical sensitivities exist. Both chemical kinetics and mass-transfer rates are thus pertinent as to whether CNF and probably many other compounds will explode or burn.

In conclusion, the work of this report period indicates that the time delay to a light output is not a reliable measure of sensitivity to explosion and other factors must be satisfied before growth-to-explosion can occur.

SECTION IV

FUTURE WORK

The ambient-pressure effect on the explosion burning mode of reaction of CNF compounds will be further evaluated. An attempt will be made to evaluate both homogeneous gas-phase initiated explosion and heterogeneous surface-reaction initiated explosion of the same compound. It is anticipated that Compound R will be suitable for this purpose since moderate ambient pressures usually result in a burning reaction, and several tests at very high ambient pressures (about 1600 psia) gave a slight indication of a second transition of events, from a burning reaction to an explosive reaction. Therefore at the more severe shock-tube condition heterogeneous surface-reaction initated explosion probably occurred.

Testing to sensitize and densensitize CNF compounds by both chemical additives and physical means also will be continued. The sensitization effects of water additives on Compound R are apparent. Ice and snow forms exhibit differences. These studies will be extended as time permits.

Because of the import of the current status of the research program, a steering committee meeting in the near future would be most advantageous to allow Edwards Air Force Base personnel to give more useful direction of the research to IITRI project personnel.

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13 ABSTRACT

In tests with Compound T, using nitrogen driven gas shocked to reflected temperatures about 1150°K, the corresponding changes in time for induction delay and sample consumption for increases in reflected-shock pressure from 500 to 1500 psia were 400 to 50 and near zero to 500 microseconds, respectively. Although the induction time decreases at higher pressure the decreased consumption rate indicates a relatively slow-burning that is an improvement in explosion sensitivity. A vaporization mechanism which appears to be inhibited by high external pressure was proposed to be a rate-controlling step in the explosion transition of most CNF type compounds that were studied to date. In additional experiments, explosions were simulated by spark discharges so as to verify certain characteristics of the light and pressure sensors as used in shock-tube sensitivity testing of cryogenic compounds. Efforts to characterize the vaporization of inert freons were only partly successful.

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	ROLE	WT	ROLE	WT	ROLE	WT
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